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IS 1448-36 (1960): Methods of test for petroleum and its products, Part 36: Sulphur mercaptans [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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Indian Standard
METHOD OF TEST FOR PETROLEUM AND ITS PRODUCTS: SULPHUR MERCAPTANS
[P : 36]

(Fifth Reprint DECEMBER 1998)

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Adapted, with permission, from the Institute of Petroleum Standard No. IP 104/53 *T*

1. SCOPE

1.1 This method is intended for the determination of the mercaptan sulphur in motor fuels and other volatile petroleum distillates containing more than 0·001 percent by weight of mercaptan sulphur.

2. OUTLINE OF THE METHOD

2.1 Hydrogen sulphide, if present, is removed from the sample, and the low boiling point mercaptans are converted into silver mercaptides by reaction with silver nitrate, the excess of which is then determined by titration with ammonium thiocyanate.

3. REAGENTS

3.1 Cadmium Sulphate Solution — containing 150 g of cadmium sulphate, $3CdSO_4 . 8H_2O$, dissolved in 120 ml of sulphuric acid (1 N) and diluted to one litre with distilled water.

3.2 Methyl or Ethyl Alcohol — 95 percent by volume, free from aldehydes.

3.3 Silver Nitrate Solution — 0·02 N, standardized by gravimetric determination of the silver as chloride.

3.4 Ferric Ammonium Alum Solution — Dissolve 40 g of ferric ammonium alum and 20 ml of dilute nitric acid (6 N) in 100 ml of distilled water and boil to remove free oxides of nitrogen. Dilute to 400 ml with distilled water.

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3.5 Ammonium Thiocyanate Solution — 0·02 N, standardized against silver nitrate solution.

4. SAMPLE

4.1 The quantity of sample to be taken depends on the mercaptan sulphur content (*see* **4.1.1**). In general the quantity of mercaptan sulphur present in the portion taken for examination should be in the range of 0·001 to 0·01 g. Suitable quantities are as follows:

Mercaptan Sulphur Content, Percent by Weight	*Quantity of Sample*
	g
Over 1·0	Less than 0·1
0·10 to 1·0	1 to 0·1
0·01 to 0·10	10 to 1
0·001 to 0·01	100 to 10

4.1.1 Samples of high mercaptan content may require dilution with methyl or ethyl alcohol so that an aliquot portion can be measured for test.

5. PROCEDURE

5.1 If the sample contains hydrogen sulphide, shake with the cadmium sulphate solution. Separate from the aqueous layer and filter.

5.2 Place a suitable measured volume of the sample or of the filtrate in a 250-ml conical flask fitted with a ground glass stopper and dilute with 15 ml of methyl or ethyl alcohol. Add 20 ml of silver nitrate solution and shake the mixture vigorously for 5 minutes. If a persistent emulsion is formed, add a further portion of methyl or ethyl alcohol. Add 2 ml of ferric ammonium alum solution and titrate the excess of silver nitrate with ammonium thiocyanate solution.

6. CALCULATION

6.1 Calculate as follows:

$$\text{Mercaptan sulphur content, percent by weight} = \frac{(V_1 - V_2)\ 0.064}{W}$$

where

V_1 = volume in ml of 0·02 N silver nitrate solution used,

V_2 = volume in ml of 0·02 N ammonium thiocyanate solution used, and

W = weight in g of sample, calculated from volume of the sample taken for the test and its specific gravity.

7. PRECISION

7.1 The precision limits of this method have not been established.

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